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**SOLID DETERGENT AND MANUFACTURE OF THE SAME**

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**SOLID DETERGENT AND METHOD FOR MANUFACTURING THE SAME**

[Kokei senjozai oyobi sono seihō]

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[There are no amendments to this patent.]

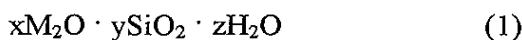
**Claims**

1. A solid detergent, characterized by being an aggregate of solid powder-form particles having an average particle diameter set to 0.05-2.0 mm, which bind to each other via water released from hydroxide that constitutes at least part thereof, thus forming a shape-retaining molding with a specific gravity of 0.7-1.4.
2. A method for manufacturing a solid detergent, characterized in that a detergent composition composed of an aggregate of powder-form solid particles with an average particle size set at 0.05-2.0 mm, at least some of which comprises hydroxide, is prepared and mixed until uniform, whereupon this mixture is then allowed to harden naturally by leaving it for a prescribed period of time in the absence of heat or pressure.

3. The method for manufacturing a solid detergent according to Claim 2, wherein the aforementioned detergent composition is prepared in a batch vessel, and after mixing until uniform, is loaded into molds in a prescribed amount, and then allowed to harden naturally in the mold.

4. The method for manufacturing a solid detergent according to Claim 2 or 3, wherein said detergent composition contains 5-50 wt% phosphoric acid alkali metal salt as a metal ion chelator, and 1-50 wt% alkali metal hydroxide and 5-50 wt% silicic acid alkali metal salt represented by chemical formula (1) below as said detergent.

[Structure 1]



(where x, y and z denote the added mole numbers, x:y is 0.25:1 to 5:1, z is an integer selected from 1, 5 and 9, and M is potassium or sodium).

5. The detergent composition according to Claim 4, wherein said silicic acid alkali metal salt is sodium metasilicate nonahydrate.

6. The method for manufacturing a solid detergent according to Claim 2 or 3, wherein said detergent composition contains 5-50 wt% phosphoric acid alkali metal salt as a metal ion chelator, and 1-50 wt% silicic acid alkali metal salt expressed by chemical formula (2) below and 5-50 wt% of at least one of sodium sulfate decahydrate and sodium carbonate decahydrate as said detergent.

7. The method for manufacturing a solid detergent according to Claim 2 or 3, wherein said detergent composition contains 5-50 wt% phosphoric acid alkali metal salt as a metal ion chelator, and 1-50 wt% silicic acid alkali metal salt represented by chemical formula (2), and 5-50 wt% of at least one of sodium sulfate decahydrate and sodium carbonate decahydrate as said detergent.

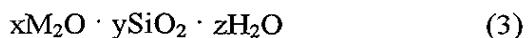
[Structure 2]



(where x, y and z denote the added mole numbers, x:y is 0.25:1 to 5:1, z is an integer selected from 0, 1, 5 and 9, and M is potassium or sodium).

8. The method for preparing a solid detergent according to Claim 7, wherein said silicic acid alkali metal salt is a silicic acid alkali metal salt represented by chemical formula (3) below.

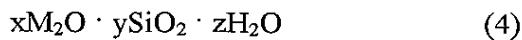
[Structure 3]



(where x, y and z denote the added mole numbers, x:y is 0.25:1 to 5:1, z is an integer selected from 1, 5 and 9, and M is potassium or sodium).

9. The method for preparing a solid detergent according to Claim 7, wherein said silicic acid alkali metal salt is a silicic acid alkali metal salt represented by chemical formula (4) below.

[Structure 4]



(where x, y and z denote the added mole numbers, x:y is 0.25:1 to 5:1, z is 0, and M denotes potassium or sodium).

Detailed explanation of the invention

[0001]

Industrial application field

The present invention relates to a solid detergent and method for manufacturing the same, which is appropriate for washing hard surfaces such as metal, glass, porcelain and plastic, and is particularly suitable from the standpoint of handling safety for automatic eating utensil washers.

[0002]

Prior art

Automatic eating utensil washers have come to be widely used for the efficient cleaning of used eating utensils in establishments such as hotels, restaurants, concessionaires, and company cafeterias. In addition to applications pertaining to foodstuffs, automatic washers have also been used in order to clean shared plastic containers, tools and other containers in manufacturing plants and processing plants. It is common knowledge that powdered detergents have been used in these types of automatic washers.

[0003]

However, when powdered detergent is used, it is necessary to dispense powdered detergent periodically into a detergent supply device that is connected to the automatic washer. Because of the possibility of spillage of the detergent powder during this time, the powder may become affixed or absorbed on the skin or the worker, which is problematic from the standpoint of sanitation control. Moreover, the aforementioned powdered detergent is introduced into the washing chamber as a liquid after being dissolved in water or warm water in the device, so that there are problems with uniform dissolution due to the different dissolution concentrations of the various components of powdered detergents.

[0004]

Liquid detergents have been offered as a substitute for powdered detergents as a means for solving these problems, but the aforementioned liquid detergents also have the danger of spillage, and because the ratio of effective component is low due to the high proportion of solvent, there is the problem that a comparatively large quantity of detergent is required. For this

reason, the unit of exchange constitutes a heavy load of material, which makes the exchange operation difficult, and also requires a great deal of space in order to accommodate the process. Moreover, thick polyethylene containers generally used for storing liquid detergent are flame resistant and decomposition resistant, which poses problems from the standpoint of waste treatment.

[0005]

Thus, solid detergents have been offered in recent years, and have been implemented in some cases. By using detergents in these systems, there are advantages from the standpoint of sanitation, in that highly concentrated detergent can be supplied at uniform concentration. In addition, a compact paper container may be used, which is advantageous from the standpoint of handling, and requires little storage space. Container treatment is also simplified. These advantages are expected to lead to an increase in demand for solid detergents.

[0006]

The aforementioned solid detergents are generally obtained by methods wherein detergent composition containing highly concentrated detergent is melted by heating, and is then cooled to solidify it. However, when the material is melted by heating, the detergent components that have poor thermal stability may undergo thermal decomposition, and in addition, energy costs for heating are high. Consequently, the development of a detergent composition that solidifies effectively while using as little heating as possible is a serious topic of investigation. In light of this state of affairs, various compositions have been offered, such as rigid solid poured detergent compositions in which two types of solid components are cast in particle form, and water is sprayed thereupon in order to cause adhesion of the solid components (Japanese Kokoku Patent No. Sho 59[1984]-4480) and detergent compositions in which water is blended intentionally in order to solidify the entire body without using a thermal fusion process (Japanese Tokuhyou Patent Application No. Hei 6[1994]-505280).

[0007]

Problems to be solved by the invention

However, with these compositions, blending of water is not completely eliminated, and when water is added in any amount, or when any of the prescribed components are added in liquid form, there is the danger that the water content will slowly spread through the entire composition. On the other hand, if the proportion of water with respect to other components is small, then preparation of the composition will be difficult.

[0008]

The present invention was developed in light of this state of affairs, and has the objective of offering a completely novel detergent composition and method for its preparation which is obtained by allowing a detergent composition composed of solid particles to solidify naturally without heat or pressure.

[0009]

Means to solve the problems

In order to achieve the above objectives, the present invention according to Claim 1 is a solid detergent, characterized in that an aggregate of solid powder-form particles having an average particle diameter set to 0.05-2.0 mm are bound to each other via water separated from the hydroxide that constitutes at least part thereof, thus forming a shape-retaining molding with a specific gravity of 0.7-1.4.

[0010]

In addition, the invention according to Claim 2 of the present invention is a method for preparing a solid detergent, characterized in that a detergent composition composed of an aggregate of powder-form solid particles with an average particle size set to 0.05-2.0 mm, at least some of which comprises hydroxide, is prepared and mixed until uniform, whereupon this mixture is then allowed to harden naturally by leaving it for a prescribed period of time in the absence of heat or pressure.

[0011]

The invention according to Claim 3 of the present invention pertains to the aforementioned solid detergent, and presents an example in which preparation of the detergent composition is carried out in a batch vessel. The inventions pertaining to Claims 4-9 of the present invention are examples wherein preferred detergent compositions are used in the preparation of the solid detergent pertaining to Claim 2 or 3.

[0012]

In the present invention, the term "hydroxide" denotes compound that contains water in molecular form.



Mode of implementation of the invention

[0013]

Modes of implementation of the present invention are described below.

[0014]

First, the solid detergent of the present invention is necessarily constituted by an aggregate of powder-form solid particles with an average particle size set at 0.05-2.0 mm, where at least some of the particles contain hydroxide. Specifically, the present invention involves the gradual release of water content contained in the hydroxide described below, where this separated water then causes binding between the solid particles that constitute the solid detergent. As a result, the entire body is allowed to solidify naturally to produce a determinate shape, so that no water or aqueous solution is blended. This is a significant characteristic of the present invention. Consequently, if the average particle size of the aforementioned solid particle aggregate is greater than 2.0 mm, then the aforementioned dissociated water alone will not uniformly solidify the entire quantity of solidified particles, and a solid detergent will not be formed. In addition, along the same lines, it is preferable for the maximum particle diameter in the aforementioned solid particle aggregate not to exceed 2.5 mm. Conversely, if the average particle diameter of the aggregate of the aforementioned solid particles is smaller than 0.05 mm, then preparation of the composition will be laborious, and the solubility of the resulting solid detergent will be poor, which will be detrimental to the advantages of the present invention.

[0015]

The aggregate of the aforementioned powder-form solid particles is composed of a detergent composition, and ordinarily, is constituted by a metal ion chelator, detergent, and various additives such as builders.

[0016]

Phosphoric acid alkali metal salts are preferred materials for use as the aforementioned metal ion chelator, where examples of the above phosphoric acid include orthophosphoric acid, polyphosphoric acid, pyrophosphoric acid, metaphosphoric acid and hexametaphosphoric acid. In addition, examples of alkali metals that are compounded therewith include sodium and potassium. Among the above phosphoric acid alkali metal salts, sodium tripolyphosphate is preferred. Other than the phosphoric acid alkali metal salt, examples of substances that may be used include ethylenediaminetetraacetic acid salts and nitrilotriacetic acid trisodium salt.

[0017]

Examples of the aforementioned detergent include alkali metal hydroxide, silicic acid alkali metal salt, sulfuric acid salts, carbonic acid salts and various other types of salts.

[0018]

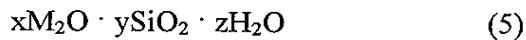
Examples of the aforementioned alkali metal hydroxide include sodium hydroxide and potassium hydroxide.

[0019]

In addition, examples of preferred silicic acid alkali metal salts are those that are represented by chemical formula (5) below, where those salts wherein x:y below is 1:1 to 3:1, and in particular, 1:1 to 2:1, are preferred from the standpoint of detergent performance and dispersibility. For example, sodium metasilicate nonahydrate is preferred. In addition, anhydrous sodium metasilicate and sodium disilicate or layered sodium silicate (SKS-6, Hoechst) may be used.

[0020]

[Structure 5]



(Where x, y and z denote the added mole numbers, x:y is 0.25:1 to 5:1, z is an integer selected from 0, 1, 5 and 9, and M is potassium or sodium.)

[0021]

In addition, preferred examples of the aforementioned sulfate include sodium sulfate decahydrate. Preferred examples of the aforementioned carbonate include sodium carbonate decahydrate, sodium bicarbonate and sodium sesquicarbonate.

[0022]

Examples of builders used for improving deterging power that may be used include anhydrous sodium sulfate, anhydrous sodium carbonate and anhydrous potassium carbonate. In addition, carboxylates such as sodium citrate, sodium gluconate, sodium tartrate, sodium malate and sodium succinate may be used. Examples of other organic builders that may be used include ethane-1,1-diphosphonate, ethane-1,1,2-triphosphonate, ethane-1-hydroxy-1,1-diphosphonate and derivatives thereof, ethanehydroxy-1,1,2-triphosphonate, ethane-1,2-dicarboxy-1,2-diphosphonate, methanehydroxyphosphonate and other phosphonic acids, 2-phosphonobutane-1,2-dicarboxylate, 1-phosphonobutane-2,3,4-tricarboxylic acid,  $\alpha$ -methylphosphonosuccinic acid and other phosphonocarboxylates, and amino acid salts formed from acids such as aspartic acid and glutamic acid.

[0023]

In addition, with the aim of providing bleaching effects, chloroisocyanurates and other chlorine-based oxidizing agents, or sodium percarbonate, sodium perborate, sodium perphthalate and other oxygen-based oxidizing agents may also be used.

[0024]

With the objective of improving the emulsion and dispersion of dirt, surfactants may also be used. Low-sudsing surfactants are preferred for use as the aforementioned surfactant, and examples that may be cited include polyoxyethylene-polyoxypropylene copolymer, phosphate esters, polyoxyethylene polyoxypropylene alkyl ether, polyoxyethylene alkylphenyl ether, polyoxyethylene sorbitan fatty acid ester, polyoxyethylene sorbitol fatty acid ester, polyethylene glycol fatty acid ester, polyoxyethylene castor oil, polyoxyethylene alkylamine, glycerol fatty acid ester, higher fatty acid alkanolamide, alkyl glycoside, and alkylamine oxide.

[0025]

In addition, examples of dispersants include polyethylene glycol, polyvinyl alcohol, polyvinylpyrrolidone and other nondissociating macromolecular substances, carboxymethylcellulose and xanthan gum.

[0026]

In addition, additives that are used in order to improve deterging effects with respect to oily dirt may also be blended, examples of which include monoethanolamine, diethanolamine, triethanolamine, N-methyl-2-pyrrolidone, diethylene glycol monoethyl ether, diethylene glycol monomethyl ether, diethylene glycol monobutyl ether, triethylene glycol monoethyl ether, triethylene glycol monobutyl ether, tetraethylene glycol monomethyl ether and tetraethylene glycol monoethyl ether.

[0027]

Bleach activators, hydrotropic agents, silicone antisudsing agents, fragrances, colorants, preservatives and other well-known components may also be blended.

[0028]

In addition, in order to improve the solidification effects of the composition, organic electrolyte macromolecular polymers may also be used. Examples of these types of organic electrolyte macromolecular polymers include polyacrylic acid, polyacrylic acid, polyitaconic acid, polycitraconic acid, polyfumaric acid, polymaleic acid, polymetaconic [transliteration] acid,

poly- $\alpha$ -hydroxyacrylic acid, polyvinylphosphonic acid, sulfonated polymaleic acid, anhydrous maleic acid-diisobutylene copolymer, anhydrous maleic acid-styrene copolymer, anhydrous maleic acid-methylvinyl ether copolymer, anhydrous maleic acid-ethylene copolymer, anhydrous maleic acid-ethylene crosslinked copolymer, anhydrous maleic acid-vinyl acetate copolymer, anhydrous maleic acid-acrylonitrile copolymer, anhydrous maleic acid-acrylic acid ester copolymer, anhydrous maleic acid-butadiene copolymer, anhydrous maleic acid-isoprene copolymer, poly- $\beta$ -ketocarboxylic acid derived from anhydrous maleic acid and carbon monoxide, itaconic acid-ethylene copolymer, itaconic acid-aconitic acid copolymer, itaconic acid-maleic acid copolymer, itaconic acid-acrylic acid copolymer, methylene malonate, itaconic acid-fumaric acid copolymer, ethylene glycol-ethylene terephthalate copolymer and vinylpyrrolidone-vinyl acetate copolymer. These substances may be used individually, or two or more types may be used in conjunction. Preferred among these substances are acrylic acid, maleic acid, methacrylic acid, fumaric acid, itaconic acid, and polymers or copolymers of these individual compounds or combinations thereof.

[0029]

The hydroxide used in order to cause natural hardening of the aforementioned powder-form solid particle aggregate may partially or entirely constitute the detergent. Examples of preferred detergent compositions that contain the above hydroxide are a detergent composition composed of 5-50 wt% ("%" below) alkali phosphate metal salt used as a metal ion chelator, 1-50% alkali metal hydroxide used as a detergent, and 5-50% of one of the silicic acid alkali metal salts represented by chemical formula (5) above.

[0030]

In addition, other examples are a detergent composition comprising 5-50% phosphoric acid alkali metal salt as metal ion chelator, 1-50% alkali metal hydroxide used as a detergent, and 5-50% of at least one of sodium sulfate decahydrate and sodium carbonate decahydrate, a detergent composition comprising 5-50% of the same phosphoric acid alkali metal salt, 1-50% anhydrous sodium silicate used as a detergent, and at least one of sodium sulfate decahydrate and sodium carbonate decahydrate, and in addition, a detergent composition comprising, in the same composition as above, a hydroxide selected from among the silicic acid alkali metal salts represented by chemical formula (5) above instead of the anhydrous sodium silicate.

[0031]

In the detergent composition used in the present invention, when the aforementioned surfactants or other liquid components (nonaqueous) are blended, it is preferable for the blending

ratio to be set at 10% or below. Thus, from the standpoint of deterging properties and emulsification/dispersion properties, it is preferable for this ratio to be set within the range of 0.1-4.0%.

[0032]

The solid detergent of the present invention can be manufactured, for example, in the manner described below using the aforementioned detergent composition. Specifically, the aforementioned detergent composition is prepared so that it has an average particle size that is within the above range, and after stirring and mixing until uniform in a batch container, the material is loaded in prescribed amounts into molds having prescribed shapes. The material is then left for a determinate period of time without heating or pressure. During this time, the water content in some or all of the detergent in the aforementioned detergent composition is released from the crystal structure in which it is incorporated, and then successively moistens each of the particles over time. For this reason, the moistened particles stick to each other, and in the end, the entire body will naturally solidify to produce a molding having shape-retention properties in the same shape as the mold. In this manner, it is possible to obtain the target solid detergent composition.

[0033]

The above mold may be the cartridge container used for supplying the detergent, or alternatively, after solidification using a prescribed form, the material can be transferred into the above cartridge container or other packaging containers.

[0034]

In addition, the natural solidification period within the aforementioned mold will depend on the total amount of composition, and for example, with 100 g of composition, solidification will occur completely within a time period of as long as 24 h, but generally in 1-5 h. In order to obtain solid detergents in the past, a process has been required wherein the entire body is liquefied by melting and cast in a mold, followed by cooling to bring about solidification, or a process has been required wherein a determinate quantity of water or aqueous solution is added to the powdered composition. By means of the present invention, however, such processes are unnecessary, and the target solid detergent can be readily obtained by simply loading and leaving the composition in a mold (container) having the prescribed shape after uniformly mixing the powdered compositions. For this reason, manufacturing costs can be lowered, and because no heating is required, there is no loss of performance of components having poor thermal stability, allowing the production of a solid detergent with excellent deterging power. Moreover, because

no water is blended in the composition, it is possible to use high-performance components that are readily hydrolyzed, but have not been used in the past, thereby allowing additional improvement in deterging power. Moreover, with the solid detergent obtained in this manner, the solid particles simply adhere to each other at the surfaces without compression, so that minute gaps remain between the particles, thereby producing a solid detergent with a low specific gravity. Specifically, the solid detergent of the present invention has a specific gravity of 0.7-1.4, which is light in comparison to that of conventional materials (specific gravity: 1.5-2). Consequently, when this solid detergent is loaded into detergent introduction devices that are connected to automatic eating utensil cleaning devices or other such equipment, the material will more readily dissolve in water or warm water relative to conventional detergents. This property is advantageous from the standpoint of ease of use.

[0035]

Moreover, in the above production method, the powder-form detergent composition is stirred and blended until uniform in a batch container, whereupon it is loaded into a mold. However, this procedure is not necessarily required, and for example, a screw-type mixing/extrusion device such as an extruder may be used wherein the various constitutive components of the aforementioned detergent composition are stirred and mixed, whereupon the material is loaded in determinate amounts into molds in a uniformly mixed condition, or is extruded into a cartridge vessel. Alternatively, the various constitutive components of the detergent composition can be directly introduced, in sequence, into the mold, and the composition can then be stirred and mixed in the aforementioned mold.

[0036]

The solid detergent of the present invention is then removed from the aforementioned mold and dispensed, or alternatively, is dispensed as an integrated body without removal from the mold. Specifically, the solid detergent that has been released and separated from the mold is wrapped in paper or film having good mold-release properties, and is then delivered to the market as a product. Alternatively, as described above, the material can be produced as a cartridge-form detergent, so that the cartridge container serves as the mold, with the solid detergent of the present invention being produced in the cartridge. Consequently, each of the containers is used as a detergent cartridge without releasing the detergent, and is sent to the market in this condition. Alternatively, the material that has solidified in a large mold can be released, removed and cut to sizes that are more readily handled, before being packaged to produce a commercial product.

[0037]

Application examples and comparative examples are presented below.

[0038]

Application Examples 1-10

Detergent compositions having the compositions indicated in Tables 1-3 below (units: parts by weight, same for subsequent tables) were prepared. The particle size of each component was adjusted so that the average particle size of the composition as a whole was 0.8 mm, and so that the maximum particle diameter did not exceed 1.5 mm. After loading each sample in the amount of 100 g into 250-mL plastic containers, the materials were left for 1 day at room temperature (20-25°C). Next, the containers containing the aforementioned detergent compositions were inverted 180°, and the degree of hardening of the content was evaluated as indicated below. In addition, the specific gravity of the contents was measured. The results are presented together in Tables 1-3.

[0039]

Evaluation of hardening

◎: Material did not break and fall out even when inverted and shaken.

O: Some of the material broke off and fell out when inverted and shaken, but the material was retained while shaking.

Δ: The material broke and fell out as a result of inversion alone.

X: All of the material broke and fell out when inverted.

[0040]

The aforementioned hardened samples were loaded into a conventional well-known solid detergent loading-type automatic eating utensil washer (JWD-6, Ishikawajima-Harima Heavy Industries), and were used in cleaning actual utensils under the conditions indicated below. The deterging power was evaluated as indicated below. The results are presented together in Tables 1-3.

[0041]

Deterging power setting conditions

- Standard cleaning cycle
- Detergent concentration: 0.12%
- Detergent temperature: 55°C
- Rinsing temperature: 80°C

Water hardness (as concentration of  $\text{CaCO}_3$ ) 70-75 ppm

[0042]

#### Evaluation method

70 parts by weight ("parts" below) margarine were introduced into an appropriate container, and after heating and melting, 15 parts powdered milk, 5 parts defatted milk and 10 parts wheat flour were added, and the mixture was uniformly dissolved. 30 parts water were then added to produce a paste for use as an evaluative stain. The aforementioned evaluative stain material was applied onto porcelain dishes with diameters of 20 cm in the amount of 8 g per dish, and was allowed to dry for 1 h at room temperature. The dishes that were stained in this manner were divided into 10 per group, and were washed under the above conditions. The condition of the cleaned stains was evaluated visually in the manner indicated below.

◎ 90% or more of the stain was removed

O: 70-90% of the stain was removed

Δ: 50-70% of the stain was removed

X: Less than 50% of the stain was removed

[0043]

Table 1

		(1) 実　　施　　例			
		1	2	3	4
②	トリポリリン酸ナトリウム	30	30	30	30
③	水　　化　　物	メタ硅酸ナトリウム・9水塩 <sup>4</sup>	10	—	—
		硫酸ナトリウム・10水塩 <sup>5</sup>	—	10	—
		炭酸ナトリウム・10水塩 <sup>6</sup>	—	—	10
⑦	無　　水　　物	水酸化ナトリウム <sup>8</sup>	42	42	42
		無水硅酸ナトリウム <sup>9</sup>	—	—	—
		無水硫酸ナトリウム <sup>10</sup>	—	—	—
		無水炭酸ナトリウム <sup>11</sup>	—	—	—
⑫	評　　価	固化の程度 <sup>13</sup>	●	●	●
		比重 <sup>14</sup>	1.4	0.9	1.0
		洗浄性 <sup>15</sup>	●	●	●

Key:

- 1 Application Example
- 2 Sodium tripolyphosphate
- 3 Hydroxide
- 4 Sodium metasilicate nonahydrate
- 5 Sodium sulfate decahydrate
- 6 Sodium carbonate decahydrate
- 7 Anhydride
- 8 Sodium hydroxide
- 9 Anhydrous sodium silicate
- 10 Anhydrous sodium sulfate
- 11 Anhydrous sodium carbonate
- 12 Evaluation
- 13 Degree of hardening
- 14 Specific gravity
- 15 Deterging properties

[0044]

Table 2

		① 実 施 例			
		6	6	7	8
②	トリポリリン酸ナトリウム	30	30	30	30
③	水 純 硫酸ナトリウム・9水塩 ④	5	5	—	10
化 物	硫酸ナトリウム・10水塩 ⑤	5	—	10	10
	硫酸ナトリウム・10水塩 ⑥	—	5	10	—
⑦	水酸化ナトリウム ⑧	—	—	—	—
無 水 物	無水硫酸ナトリウム ⑨	—	—	30	20
	無水硫酸ナトリウム ⑩	—	—	—	10
	無水炭酸ナトリウム ⑪	—	—	—	20
⑫	固 化 の 程 度 ⑬	●	●	●	●
	比 重 ⑭	1.4	1.4	1.0	1.0
	洗 浸 性 ⑮	●	●	●	●

Key:

- 1 Application Example
- 2 Sodium tripolyphosphate
- 3 Hydroxide
- 4 Sodium metasilicate nonahydrate
- 5 Sodium sulfate decahydrate
- 6 Sodium carbonate decahydrate
- 7 Anhydride
- 8 Sodium hydroxide
- 9 Anhydrous sodium silicate
- 10 Anhydrous sodium sulfate
- 11 Anhydrous sodium carbonate
- 12 Evaluation
- 13 Degree of hardening

14 Specific gravity  
 15 Deterging properties

[0045]

Table 3

	(1)	実施例9	実施例10
(2)	トリポリリン酸ナトリウム	20	—
(3)	エチレンジアミンテトラ酢酸ナトリウム	—	20
(4) 化 物	メク基酸ナトリウム・9水塩 <sup>(5)</sup>	50	—
	硫酸ナトリウム・10水塩 <sup>(6)</sup>	—	30
	炭酸ナトリウム・10水塩 <sup>(7)</sup>	—	—
(8) 無 水 物	水酸化ナトリウム <sup>(9)</sup>	30	40
	無水硫酸ナトリウム <sup>(10)</sup>	—	—
	無水硫酸ナトリウム <sup>(11)</sup>	—	10
	無水炭酸ナトリウム <sup>(12)</sup>	—	—
(13) 評 価	固化的程度 <sup>(14)</sup>	○	○
	比重 <sup>(15)</sup>	0.7	0.7
	洗浄性 <sup>(16)</sup>	○	○

Key:

- 1 Application Example
- 2 Sodium tripolyphosphate
- 3 Sodium ethylenediaminetetraacetate
- 4 Hydroxide
- 5 Sodium metasilicate nonahydrate
- 6 Sodium sulfate decahydrate
- 7 Sodium carbonate decahydrate
- 8 Anhydride
- 9 Sodium hydroxide
- 10 Anhydrous sodium silicate
- 11 Anhydrous sodium sulfate

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- 12 Anhydrous sodium carbonate
- 13 Evaluation
- 14 Degree of hardening
- 15 Specific gravity
- 16 Deterging properties

[0046]

Application Examples 11-22

Detergent compositions having the compositions indicated in Tables 4-6 below were prepared. The settings for the maximum particle diameter and the average particle diameter of the entire composition were the same as in the above application examples. The degree of hardening, specific gravity and deterging properties were evaluated in the same manner as in the above application examples, and the results are presented together in Tables 4-6.

[0047]

Table 4

		(1) 実　　施　　例			
		1.1	1.2	1.3	1.4
(2)	金属イオン封鎖剤	トリポリリン酸ナトリウム (3)	—	—	5 50
		エチレンジアミンテトラ酢酸ナトリウム (4)	20	—	—
		ニトリロ3酢酸3ナトリウム (5)	—	20	—
	メタ珪藻ナトリウム・9水塩 (6)	30	30	30 30	30
	水酸化ナトリウム (7)	35	—	42 14	
	無水珪藻ナトリウム (8)	—	35	—	—
	無水硫酸ナトリウム (9)	—	—	23 6	
(10)	評価	硬化の程度 (11)	●	●	● ●
		比重 (12)	0.8	1.2	0.9 0.9
		洗浄性 (13)	●	●	● ●

Key: 1 Application Example  
 2 Metal ion chelator  
 3 Sodium tripolyphosphate  
 4 Sodium ethylenediaminetetraacetate  
 5 Trisodium nitrilotriacetate  
 6 Sodium metasilicate nonahydrate  
 7 Sodium hydroxide  
 8 Anhydrous sodium silicate  
 9 Anhydrous sodium sulfate  
 10 Evaluation  
 11 Degree of hardening  
 12 Specific gravity  
 13 Deterging properties

[0048]

Table 5

		(1) 実　　験　　例				
		15	16	17	18	
(2)	金属イオン封鎖剤	トリポリリン酸ナトリウム (3)	20	20	50	20
		エチレンジアミンテトラ酢酸ナトリウム (4)	---	---	---	---
		ニトリロ3酢酸3ナトリウム (5)	---	---	---	---
	メタ硅酸ナトリウム・9水塩 (6)		5	50	49	11
	水酸化ナトリウム (7)		50	21	1	50
	無水硅酸ナトリウム (8)		---	---	---	---
	無水硫酸ナトリウム (9)		25	9	—	19
(10)	硬化の程度 (11)		○	○	○	○
	比重 (12)		1. 3	0. 8	0. 9	1. 1
	洗浄性 (13)		○	○	○	○

Key: 1 Application Example  
 2 Metal ion chelator  
 3 Sodium tripolyphosphate  
 4 Sodium ethylenediaminetetraacetate  
 5 Trisodium nitrilotriacetate  
 6 Sodium metasilicate nonahydrate  
 7 Sodium hydroxide  
 8 Anhydrous sodium silicate  
 9 Anhydrous sodium sulfate  
 10 Evaluation  
 11 Degree of hardening  
 12 Specific gravity  
 13 Cleaning

[0049]

Table 6

		(1) 実施例				
		19	20	21	22	
(2)	金属イオン対 緩衝剤	トリポリリン酸ナトリウム (3)	30	30	30	30
		エチレンジアミンテトラ酢酸ナトリウム (4)	---	---	---	---
		ニトリロ3フェニルアミノトリウム (5)	---	---	---	---
	メタ硅酸ナトリウム・9水塩 (6)	20	20	20	20	
	水酸化ナトリウム (7)	35	35	35	35	
	無水硫酸ナトリウム (8)	---	---	---	---	
	無水硫酸ナトリウム (9)	14.9	11	10	5	
	界面活性剤 (ポリアルキレン重合物) (10)	0.1	4	5	10	
(11)	硬化の程度 (12)	○	○	○	○	
	比重 (13)	1.0	1.0	1.0	0.9	
	洗浄性 (14)	○	○	○	○	

Key:

- 1 Application Example
- 2 Metal ion chelator
- 3 Sodium tripolyphosphate
- 4 Sodium ethylenediaminetetraacetate
- 5 Trisodium nitrilotriacetate
- 6 Sodium metasilicate nonahydrate
- 7 Sodium hydroxide
- 8 Anhydrous sodium silicate
- 9 Anhydrous sodium sulfate
- 10 Surfactant (polyalkylene polymer)
- 11 Evaluation
- 12 Degree of hardening
- 13 Specific gravity
- 14 Cleaning

[0050]

Application Examples 23-29, Comparative Examples 1 and 2

Particles with various compositions were prepared which had average compositional particle diameters and maximum particle diameters indicated by the values in Tables 7 and 8 below. With this exception, the target detergent compositions were obtained in the same manner as in Application Example 1 above. The degree of hardening, specific gravity and deterging properties were evaluated in the same manner as in the above application examples, and the results are compiled in Tables 7 and 8.

[0051]

Table 7

	① 比較例1	実② 比 例			
		23	24	25	26
③ 組成物の平均粒子径 (mm)	0. 03	0. 05	0. 1	0. 3	0. 8
④ 最大粒子径 (mm)	0. 5	1. 5	1. 5	1. 0	1. 5
⑤ ⑥ ⑦ ⑧	⑤ ⑥ ⑦ ⑧	○	○	○	○
		1. 5	1. 4	1. 3	1. 2
		△	○	○	○

Key:

- 1 Application Examples
- 2 Comparative Example 1
- 3 Average compositional particle diameter (mm)
- 4 Maximum particle diameter (mm)
- 5 Evaluation
- 6 Degree of hardening
- 7 Specific gravity
- 8 Deterging properties

[0052]

Table 8

	(1) 対 比 例			(2) 比較例 2	
	27	28	29		
(3) 錠成物の平均粒子径 (mm)	1.2	1.5	2.0	2.5	
(4) 最大粒子径 (mm)	2.0	2.0	2.5	3.0	
(5)	硬化の程度 (6)	○	○	△	×
	比重 (7)	1.0	0.95	0.9	0.86
	洗浄性 (8)	○	○	○	△

Key:

- 1 Application Example
- 2 Comparative Example 2
- 3 Average compositional particle diameter (mm)
- 4 Maximum particle diameter (mm)
- 5 Evaluation
- 6 Degree of hardening
- 7 Specific gravity
- 8 Deterging properties

[0053]

#### Comparative Example 3

The detergent composition having the composition indicated below was heated and melted at 55-65°C, and 100 g of the melted liquid were loaded into the same container as in the above application examples. After casting and hardening in this manner, the product, which was used as Comparative Example 3, was evaluated in terms of hardening, specific gravity and deterging properties in the same manner as in the above application examples. The results are presented in Table 9.

[0054]

#### Composition of Comparative Example 3

Sodium hydroxide	40 parts
Sodium tripolyphosphate	30 parts
Sodium orthophosphate	5 parts

Water 25 parts  
Total 100 parts

[0055]

Table 9

		1 比較例3
2 評 価	硬化の程度 比較 4	3 ○ 1.7
	洗浄性 5	△

Key: 1 Comparative Example 3  
2 Evaluation  
3 Degree of hardening  
4 Specific gravity  
5 Deterging properties

[0056]

#### Effect of the invention

With the solid detergent of the present invention described above, the surfaces of the solid particles simply bind to each other, so that a light solid detergent with a specific gravity of 0.7-1.4 is obtained due to the minute gaps remaining between the particles. Consequently, the product can dissolve more readily than conventional solid detergents, which provides advantages in terms of ease of use. By means of the method for preparing the solid detergent of the present invention, the aforementioned solid particle aggregate naturally hardens without heat or pressure due to the water content released by the hydroxide constituting at least part of the detergent composition in the solid particle aggregate. As a result, a solid detergent is obtained, so that it is unnecessary to use a process in which the entire composition is heated and melted to produce a liquid, followed by casting and solidification by cooling. It is also unnecessary to use a process wherein a determinate quantity of water or liquid is added to the powder-form composition. The target solid detergent can thus be readily obtained by simply loading the powder-form composition into a prescribed mold (container) in a uniformly mixed condition, and then allowing the composition to stand. As a result, manufacturing costs can be reduced. In addition, a solid detergent composition with excellent detergent power can be offered, because no heating is

required, and there is thus no loss in performance of components having poor thermal stability. Moreover, no water is blended in the composition, thus allowing the use of high-performance hydrolysable components that have been impossible to use in the past. The deterging power of the composition can thus be additionally improved.